Study of tribochemical processes on hard disks using Photoemission Electron Microscopy

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INTRODUCTION

With increasing storage density of magnetic media reaching 10 Gb/in^2 and increasingly smaller spacing between slider and hard disk surface approaching pseudo-contact, the interface between slider and disk becomes more and more important. Due to the small spacing there is a strong interaction between the hard carbon overcoat of the disk, the lubricant, and the surface of the slider, which often is also coated by amorphous hard carbon. Optimization of the tribological properties can only be obtained by considering the whole system and the interactions of the individual components. Tribochemistry plays a more important role for smaller spacings and thinner lubricants and protective overcoats.

We have applied Near Edge X-ray Absorption Fine Structure (NEXAFS) spectroscopy and Photoemission Electron Microscopy (PEEM) to study the elemental and chemical structure and modification of disks exposed to various forms of wear.

WEAR TESTS OF DISKS

Three different sets of disks were tested under various conditions in vacuum and in air.

Set1:

95mm disks coated with 7.5 nm sputter deposited hydrogenated amorphous hard carbon CH_x with a low hydrogen content of 5% were exposed to a continuous drag test in ultra-high vacuum. The number of cycles until failure was monitored. Uncoated sliders (Al_2O_3/TiC) and sliders coated with sputter deposited CH_x were used at a speed of 0.2 m/s and a load of 40 mN. The disks were lubricated with 0.85nm of either a perfluoropolyether (Z-DOL), a cyclic phosphazene lubricant (X-1P), or a mixture of 94% Z-DOL and 6% X1P [1]. The tribological results are published in detail in [2]. The time between the tribotesting and the PEEM studies was several weeks.

Set2:

Supersmooth disks were coated with 5 nm hydrogen-free, amorphous hard carbon formed by cathodic arc deposition. The disks were lubricated with 0.85 nm Z-DOL. The disks were worn in a continuous drag test at a speed of 0.2 m/s and a load of 30 mN in UHV. Two kinds of sliders were used: uncoated sliders (Al_2O_3/TiC) and sliders coated with sputter deposited CH_x. The time between the wear test and the microscopy study was kept below 2h to prevent lubricant from flowing back into the wear tracks. Details of the wear test are described in [2, 3].

Set 3:

95mm disks coated with 15 nm sputter deposited CH_xN_y without lubricant were exposed to a continuous drag test under ambient conditions. Sliders coated with sputter deposited CH_x at a drag speed of 0.08 m/s and a load of 30mN were used for the test. The tests were run for durations from 30s to 2h, and the friction force was recorded during the wear. These disks were specifically worn for PEEM studies of disk and slider wear. Results on the wear of the sliders will be published in [4].

EXPERIMENTAL CONDITIONS FOR PEEM AND NEXAFS STUDIES OF WEAR TRACKS ON DISKS

PEEM microscopy was applied to study elemental composition and chemical state of samples using NEXAFS spectroscopy with high spatial resolution. A two-lens, electrostatic microscope operating at a nominal voltage of 10 kV was used for these studies. The microscope has a spatial resolution of 200 nm, and it is described in detail in [5]. We used the undulator beamline 8.0 equipped with a spherical grating monochromator having three different gratings to cover the energy range from 200-1500 eV with a resolving power of $E/\Delta E$=10,000.

In all cases it was checked that the X-ray radiation does not damage the samples by comparing successive NEXAFS scans.

RESULTS

Set 1:

Local NEXAFS spectra were acquired outside and inside the wear tracks. The spectra outside the wear tracks showed the carbon π^* resonance at 285 eV and the broad σ shape resonance around 300 eV [6]. It was observed that a strong new peak is present in the spectrum of the wear track at 289.0 eV and a smaller peak at 290.9 eV. These peaks can be attributed to the formation of new carbon-oxygen bonds probably in carboxylic (289.0 eV) and carbonate form (290.9 eV) [6]. This demonstrates that the lubricant has been altered chemically and oxidation has occurred during or after the wear. The chemical modifications are the smallest for the mixed lubricant tested with a carbon coated slider. These findings agree with the results of the wear test which showed better tribological behavior for coated versus uncoated sliders and mixed lubricant versus the single components. Oxygen K edge spectra showed a considerable increase in oxygen concentration in the wear tracks for all cases, confirming the oxidation of the lubricant.

Set 2:

Figure 1c shows a PEEM image of a wear track produced by a coated slider. The scratch is caused by one of the rails of the slider. The image was taken at a photon energy of 280 eV which is below the carbon K edge, therefore the image contrast is mainly topological. Local NEXAFS spectra were taken in the undamaged area of the disk, in the wear track caused by the rail, and in the area between the rails. Figure 1a shows the carbon K edge spectra and Figure 1b the fluorine K edge spectra.

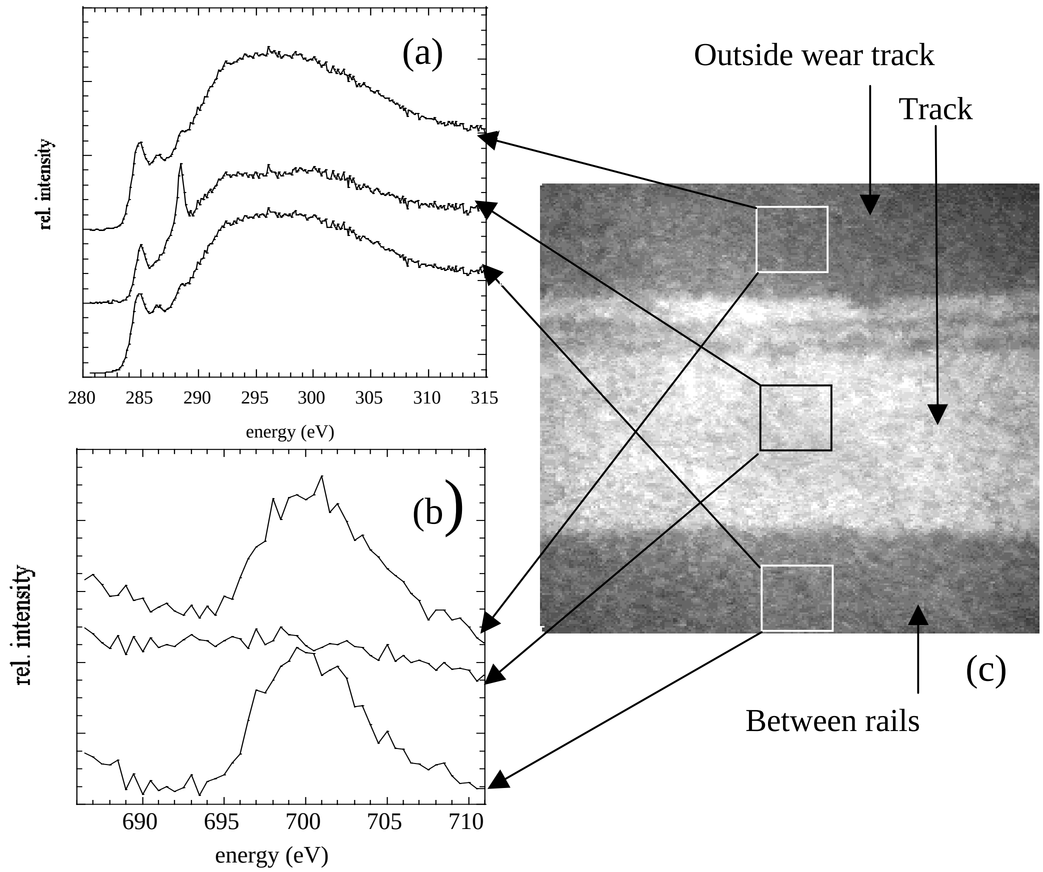

Figure 1: (a) Local carbon K edge NEXAFS spectra of indicated areas. (b) Local fluorine K edge NEXAFS spectra of indicated areas. (c) PEEM image of a wear track caused by a coated slider on a Z-DOL lubricated disk taken at a photon energy of 280 eV. The field of view is 75µm.

From the spectra we can see that the carbon overcoat/lubricant are identical in the undamaged area and between the rails. In the wear track caused by one of the rails the fluorine is almost completely removed. The removal of fluorine in the wear tracks was only observed when the time between the wear test and the PEEM studies was very short (below a few hours). In any other case when the time between the PEEM or NEXAFS study was many days the fluorine signal was identical inside and outside the wear tracks. This confirms that the lubricant is flowing back into the wear track and covers it after a while. The carbon spectrum shows a new peak around 289.0eV which can again be attributed to the formation of carboxylic bonds [6] and a small reduction of the total carbon signal. Whereas the modified carbon is still visible long after the wear test the fluorine reduction is only observed during a short period of several hours after the wear. It seems that the chemically modified carbon remains in the wear track and is later covered by unmodified lubricant flowing over the track from the side. Since the probing depth of NEXAFS and PEEM is about 10 nm for carbon materials, we see a superposition signal from the modified lubricant, the hard carbon overcoat, and unmodified lubricant if it had time to flow back into the track.

Set 3:

Local NEXAFS spectra at the carbon K edge were taken in the wear track and outside the wear track. Peaks were observed at 285 eV corresponding to the carbon π^* resonance, around 286.3 eV corresponding to C=N bonds, and a broad peak around 300 eV (σ shape resonance of carbon). It was found that the shape of the spectra is identical but the intensity in the wear track is reduced by about 40%. This indicates that the chemical state of the CH_xN_y has not been changed by the wear but some CH_xN_y has been removed.

SUMMARY

(1) Lubricated disks exposed to wear tests show chemical modifications of the lubricant when the disks failed the wear test. These modifications consist of oxidation of carbon in the form of mainly carboxylic bonds, and a reduction of the fluorine and carbon content. The chemical modifications occur in tracks that are typically 10-50 μm wide.

(2) Non-modified lubricant covers the tracks and the oxidized lubricant after a certain time period that is determined by the diffusion constant of the lubricant. The degraded, oxidized lubricant stays in the tracks over long periods of time (many weeks) and does not seem to move out of the tracks.

(3) Unlubricated disks show no chemical modifications of the hard carbon overcoat in the wear tracks, just a reduction of the overcoat thickness.

(4) The chemical modifications are correlated to the tribological behavior of the disks.

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